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(54) Title: POLYOLEFIN PRESSURE SENSITIVE ADHESIVE TAPE WITH AN IMPROVED PRIMING LAYER

(57) Abstract: The present invention is directed to a primer composition. The primer composition comprises a maleated thermoplastic elastomer, a non-halogenated polyolefin, and a resin. In some embodiments, the primer comprises a crosslinking agent, for example 2,4-bis(trichloromethyl)-6-4'-methoxyphenyl-sym-triazine. In other embodiments, the primer comprises an epoxy. The invention is also directed to a tape comprising a substrate, a primer coated on at least one surface of the substrate, the primer comprising a maleated rubber, a non-halogenated polyolefin and a resin, and a pressure sensitive adhesive coated on the primer.

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POLYOLEFIN PRESSURE SENSITIVE ADHESIVE TAPE WITH AN IMPROVED PRIMING LAYER

Field of the Invention

5 This invention relates to a primer that improves adhesion of a pressure sensitive adhesive to a tape backing.

Background of the Invention

10 In the field of pressure sensitive adhesives, predictable control over mechanical and process properties is desirable so that such adhesives can be tailored for specific, highly demanding end use applications such as packaging, medical, and masking tapes. These applications require a proper balance of properties, and this balance changes with each end use.

15 Natural and synthetic rubbers were among the earliest polymers to provide a reasonable balance of the properties required for satisfactory pressure sensitive adhesive performance. However, those skilled in the art recognized that ethylenically unsaturated groups had to be eliminated from the polymer backbone to improve the oxidative stability of the adhesives. This became possible with the discovery of the catalytic activity of Ziegler-Natta (ZN) coordination systems toward α -olefin monomers and the subsequent
20 production of high molecular weight polymers. Homopolymers of the C_6 to C_{10} α -olefins were naturally tacky, had low toxicity, aged well, and were environmentally stable, chemically inert, resistant to plasticizer migration, and relatively inexpensive. These characteristics made them good candidates for pressure sensitive adhesive applications. However, their poor cohesive strength meant that they lacked the shear adhesion necessary
25 for high performance pressure sensitive adhesives.

 Poly- α -olefins adhesive compositions comprising mixtures of α -olefin polymers and copolymers are known. See, e.g., U.S. Pat. Nos. 3,542,717 and 3,635,755. However, such compositions proved not to have sufficient shear adhesion at elevated temperatures to be suitable for demanding applications such as, for example, masking tapes. This high
30 temperature shear problem was solved in U.S. Pat. No. 5,112,882. That patent describes a radiation curable, α -olefin homo- or copolymer pressure sensitive adhesive composition

based primarily on α -olefins. That composition provides adhesive films with a superior balance of peel and shear performance.

Poly- α -olefin pressure sensitive adhesives adhere in a similar fashion to many surfaces, including low energy surfaces, and have proven to be good at not damaging the surfaces of sensitive surfaces (e.g., anodized aluminum as is used in certain aircraft parts). However, poly- α -olefin pressure sensitive adhesives have not been widely used for certain high performance applications because of adhesive transfer to the surface. This adhesive transfer is an anchorage failure rather than a cohesive failure of the adhesive (i.e., the adhesive does not remain bonded to the backing but instead transfers en masse to the protected surface when the tape is removed). This transfer most often occurs during removal of the pressure sensitive adhesive from an adherend at a high temperature (e.g., 165 °C or greater), from an adherend that has been heated to a high temperature and then allowed to cool, and/or under some very low rate peel conditions.

One of the most stringent applications for any pressure sensitive adhesive is that of a high temperature masking tape, often used in industry during painting and detailing processes. Such masking tapes must protect surfaces during bake cycles of up to an hour at temperatures between about 200 °C and about 220 °C, generally about 215 °C, yet remove cleanly from the surface thereafter.

In addition to high temperature industrial masking applications, masking of aircraft during painting procedures has become quite challenging. Tapes presently used to mask aircraft during painting have proven to be ineffective in the presence of high boiling solvents used in low-volatile organic compound (VOC) paints. Historically, paints containing VOCs such as toluene, heptane, mineral spirits, and methyl ethyl ketone, have been used during various steps of the aircraft painting process. However, governmental entities have begun to heavily regulate the use of such VOCs, making questionable the continued use of paints containing them. A masking tape able to withstand prolonged exposure to the high boiling solvents presently being used in paint formulations has yet to be demonstrated.

A poly- α -olefin pressure sensitive adhesive tape construction in which the adhesive does not transfer to the protected surface is highly desirable. Additional benefits would be achieved if such a tape construction could be used for masking applications where low-VOC solvents are to be used.

Summary of the Invention

The present invention is directed to a primer composition. The primer composition comprises a maleated thermoplastic elastomer, a non-halogenated polyolefin, and a resin. In some embodiments, the primer comprises a crosslinking agent, for example 2,4-bis(trichloromethyl)-6-4'-methoxyphenyl-sym-triazine. In other embodiments, the primer comprises an epoxy.

The invention is also directed to a tape comprising a substrate, a primer coated on at least one surface of the substrate, the primer comprising a maleated rubber, a non-halogenated polyolefin and a resin, and a pressure sensitive adhesive coated on the primer.

Detailed Description of the Invention

Pressure Sensitive Adhesive

Any suitable pressure sensitive adhesive composition can be used for a tape article of this invention. The pressure sensitive adhesive component of the tape article can be any material that has pressure sensitive adhesive properties. Pressure sensitive adhesives are well known to one of ordinary skill in the art to possess properties including the following: (1) aggressive and permanent tack, (2) adherence to an adherend with no more than finger pressure, (3) sufficient ability to hold onto an adherend, and (4) sufficient cohesive strength to be removed cleanly from the adherend. Furthermore, the pressure sensitive adhesive component can be a single pressure sensitive adhesive or the pressure sensitive adhesive can be a combination of two or more pressure sensitive adhesives.

Pressure sensitive adhesives useful in the present invention include, for example, those based on natural rubbers, synthetic rubbers, styrene block copolymers, polyvinyl ethers, poly (meth)acrylates (including both acrylates and methacrylates), polyolefins, and silicones.

In certain embodiments, the pressure sensitive adhesive is a polyolefin based pressure sensitive adhesive. In specific embodiments, the pressure sensitive adhesive is a poly- α -olefin pressure sensitive adhesive. The poly- α -olefin comprises one or more monomer units derived from an α -olefin monomer that is a liquid at standard temperature and pressure. The monomer may be a $C_5 - C_{30}$ α -olefin, for example a $C_6 - C_{20}$ α -olefin. In specific embodiments, the monomer is a $C_6 - C_{12}$ α -olefin. Such monomers optionally can be substituted with conventional substituents (i.e., those that do not interfere with the

polymerization of these monomers or with the desired properties of the polymer produced therefrom). Such an olefin can either be linear or branched (i.e., comprising one or more side chains). Common examples include 1-butene, 1-pentene, 1-hexene, 1-heptene, 1-octene, 1-nonene, 1-decene, 1-undecene, 1-dodecene, 1-hexadecene, 1-octadecene, and 4-methyl-1-pentene. Specific embodiments utilize α -olefin monomers of 1-hexene or 1-octene. Blends of one or more of these monomers plus a lower (i.e., $C_2 - C_4$) 1-alkene are also within the scope of the present invention. A full description of specific α -olefin polymers can be found in U.S. Pat. No. 5,112,882.

The α -olefin polymer can be a homopolymer or a random co, ter, or tetra polymer. It can also comprise blocks of homopoly (α -olefin) interspersed with monomer units derived from various other copolymerizable monomers. Examples of potentially useful copolymerizable monomers include polyenes such as, for example, the $C_6 - C_{14}$ α , ω -dienes, conjugated dienes, trienes, terpenes, and alkenyl-norbornenes.

The above-described monomers can be polymerized, either in bulk or in one or more inert solvents, in the presence of a catalyst system over a wide range of temperatures, e.g., about 0° to about 140° C, such as about 30° to about 90° C. The amount of catalyst used may be in the range of about 0.1 to about 5 g per kg of monomer. Useful catalyst systems include standard ZN catalyst systems, ZN catalyst systems where the transition metal compound is supported (e.g., on a $MgCl_2$ powder), and Kaminsky-Ewen catalyst systems. All three catalyst systems are well known by those familiar with α -olefin polymerizations. Because the particular catalyst system used does not affect either the primer composition or the overall tape construction of the present invention, they are not discussed in detail here.

In certain embodiments, the polymer has a glass transition temperature in the range of about -70° to about 0° C, generally in the range of about -60° to about -20° C; an inherent viscosity in the range of 0.4 to 9.0 dL/g, for example 0.5 to 6.0 dL/g, in a specific example 1.5 to 4.0 dL/g; and a number average molecular weight in the range of 5,000 to 50,000,000, for example 500,000 to 5,000,000.

Addition of one or more tackifying resins to the poly- α -olefin can improve tack, lower viscosity, improve coatability, improve peel adhesion, and enhance shear adhesion (with no concomitant loss of peel adhesion). Where a tackifying resin is used, it can be present in an amount from more than 0 to 150 parts by weight per 100 parts of polymer.

Potentially useful tackifying resins include terpene resins and those derived from polymerization of C₅ to C₉ unsaturated hydrocarbon monomers. Examples of commercially available resins based on a C₅ olefin fraction of this type are those sold under the tradenames Wingtack 95 and Wingtack 115 tackifying resins (commercially available from Goodyear Tire and Rubber Co., Akron, Ohio); Regalrez 1078 and Regalrez 1126 (commercially available from Hercules Chemical Co. Inc. Wilmington, Del.); Arkon P115 (commercially available from Arakawa Forest Chemical Industries, Chicago, Ill.); and Escorez resins (commercially available from Exxon Chemical Co. Houston, Tex.). Suitable terpene resins include terpene polymers, such as polymeric resinous materials obtained by polymerization and/or copolymerization of terpene hydrocarbons such as the alicyclic, monocyclic, and bicyclic monoterpenes and their mixtures. Commercially available terpene resins include those sold under the tradename Zonarez B-series and 7000 series terpene resins (commercially available from Arizona Chemical Corp. Wayne, N.J.). The tackifying resin can contain ethylenic unsaturation; however, saturated tackifying resins are preferred for those applications where resistance to oxidation is important. This discussion of tackifiers is not intended to be comprehensive because they are not the subject of the present invention.

Minor amounts of additives also can be included in the polymer composition to provide adhesives for special end uses. Such additives can include pigments, dyes, plasticizers, fillers, stabilizers, UV radiation absorbers, antioxidants, processing oils, and the like. The amount of additive(s) used can vary from 0.1 to 50 weight percent, depending on the end use desired. Any additive(s) used generally do not significantly absorb radiation near the wavelength of maximum absorption of any photocrosslinker included in the polymer composition.

The pressure sensitive adhesive composition may also include a crosslinking agent that is activated by actinic radiation, typically after the pressure sensitive adhesive is coated. Suitable photocrosslinking agents include, but are not limited to, (a) aldehydes, such as benzaldehyde, chromophore-substituted acetaldehyde, and their substituted derivatives; (b) ketones, such as acetophenone, benzophenone, and their substituted derivatives; (c) quinones, such as the benzoquinones, anthraquinone, and their substituted derivatives; (d) thioxanthenes, such as 2-isopropylthioxanthone and 2-dodecylthioxanthone; and (e) certain chromophore-substituted vinyl halomethyl-sym-

triazines, such as 2,4-bis(trichloromethyl)-6-4'-methoxyphenyl-s-triazine and 2,4-bis(trichloromethyl)-6-3',4'-dimethoxyphenyl-s-triazine. (Because many such triazines produce HCl upon activation, the addition of a basic compound to the polymeric composition can be beneficial.) Photoactive crosslinking agent can be present in a range
5 from about 0.005 to about 2% (by wt.), for example from about 0.01 to about 0.5% (by wt.), and in specific examples from about 0.05 to 0.15% (by wt.), of the polymer.

Primer

The primer layer comprises a thermoplastic elastomer that has been maleated, for
10 example by grafting or copolymerization with maleic anhydride. The term "maleated" means that the thermoplastic elastomer is modified, for example with maleic acid or maleic anhydride, to contain an average of one or more carboxyl groups. The thermoplastic elastomer may be a block copolymer comprising one or more polystyrene blocks and the thermoplastic elastomer may be a rubber, such as a styrene-ethylene-
15 butene-styrene (S-EB-S) type block copolymer elastomer. Examples of suitable thermoplastic elastomers include those sold under the tradename KRATON, commercially available from Kraton Polymers, Inc., Houston, TX. A specific example of a maleated rubber suitable for the present invention is sold under the tradename KRATON FG 1901X, commercially available from Kraton Polymers, Inc., Houston, TX. Other
20 examples of maleated thermoplastic elastomers include those sold under the tradenames FUSABOND MF-416D and FUSABOND MN-493D, commercially available from E.I. DuPont de Nemours Co., Wilmington, DE.

The primer additionally comprises a resin. The resin may be any resin with a glass transition temperature high enough to raise the glass transition temperature of the
25 elastomer portions of the primer and to cause the primer to have a significantly increased overall glass transition temperature. Generally, the glass transition temperature of the resin is between about 0°C and about 100°C, for example between 25°C and 100°C. In specific examples, the glass transition temperature is between about 60°C and about 80°C. The resin may be, for example, hydrocarbon resin. Generally, useful resins have low
30 molecular weights. An example of a resin suitable for the present invention are those sold under the tradename REGALREZ 1139, commercially available from Hercules Inc., Wilmington, DE.

The primer additionally comprises a polyolefin polymer. For example, the polymer may comprise a monomer such as a $C_2 - C_{30}$ α -olefin monomer, for example a $C_3 - C_{20}$ α -olefin, and in specific examples a $C_6 - C_{12}$ α -olefin. Such monomers optionally can be substituted with conventional substituents (i.e., those that do not interfere with the polymerization of these monomers or with the desired properties of the polymer produced therefrom). Generally, these polyolefins are non-halogenated. It is well known to add halogenated polyolefins to a primer. It was surprisingly found that a non-halogenated polyolefin excels in tape constructions of the present invention. Such an olefin can either be linear or branched (i.e., comprising one or more side chains). Common examples include 1-propene, 1-butene, 1-pentene, 1-hexene, 1-heptene, 1-octene, 1-nonene, 1-decene, 1-undecene, 1-dodecene, 1-hexadecene, 1-octadecene, 4-methyl-1-pentene and combinations thereof. Particular examples of α -olefin monomers include 1-hexene and 1-octene. Blends of one or more of these monomers plus a lower (i.e., $C_2 - C_4$) 1-alkene are also within the scope of the present invention. The polyolefin may also be a copolymer. A specific example of a suitable polyolefin is a polyhexene made as described in Example 4 of U.S. Patent No. 5,644,007 to Davidson, et al. and a polyoctene made as described in Example 3 of U.S. Patent No. 5,644,007 to Davidson. Additional examples include those commercial products sold under the trade names ENGAGE from DuPont Dow Elastomers L.L.C., Wilmington, DE, and ACHIEVE from Exxon Chemical Co., Houston, TX.

The primer may additionally comprise a crosslinking agent. Suitable crosslinkers include materials activated by ultraviolet-light (photocured) or exposure to heat. The primer may then be cured using a source of actinic radiation of sufficient energy (i.e., wavelength range) to generate free radicals when incident upon the particular photoactive crosslinking agent selected for use in the composition. Generally, a useful wavelength range for the photoactive crosslinking agents disclosed above is 400 to 250 nm. The radiant energy in this range of wavelengths required to crosslink the adhesive film of the invention is about 100 to about 1500 millijoules/cm² and in specific embodiments, the radiant energy is about 200 to about 800 millijoules/cm². Examples of a photocure process are disclosed in U.S. Pat. Nos. 4,181,752 and 4,329,384.

Examples of suitable photocrosslinking agents for use in the compositions of the invention include, but are not limited to: aldehydes, such as benzaldehyde, acetaldehyde,

and their substituted derivatives; ketones such as acetophenone, benzophenone and their substituted derivatives; quinones such as the benzoquinones, anthraquinone and their substituted derivatives; thioxanthenes such as 2-isopropylthioxanthone and 2-dodecylthioxanthone; and certain chromophore-substituted vinyl halomethyl-sym-triazines. In specific embodiments, the crosslinking agent is 2,4-bis(trichloromethyl)-6-4'-methoxyphenyl-sym-triazine. Such chromophore-substituted vinyl halomethyl-sym-triazines may be prepared by the co-trimerization of an aryl nitrile with trichloroacetonitrile in the presence of HCl gas and a Lewis acid such as AlCl_3 , AlBr_3 , etc. [Bull. Chem. Soc. Japan, Vol. 42, page 2924 (1969)].

The primer additionally may comprise an epoxy. Epoxy resins useful in the primer compositions of the invention are any organic compounds having at least one oxirane ring. Epoxy resins are polymerizable by a ring opening reaction. Such materials, broadly called epoxides, include both monomeric and polymeric epoxides and can be aliphatic, alicyclic, heterocyclic, cycloaliphatic, or aromatic and can be combinations thereof. They can be liquid or solid or blends thereof. These materials generally have, on the average, at least two epoxy groups per molecule and are also called "polyepoxides." The polymeric epoxides include linear polymers having terminal epoxy groups (for example, a diglycidyl ether of a polyoxyalkylene glycol), polymers having skeletal oxirane units (for example, polybutadiene polyepoxide), and polymers having pendent epoxy groups (for example, a glycidyl methacrylate polymer or copolymer). The molecular weight of the epoxy resin may vary from about 74 to about 100,000 or more. Mixtures of various epoxy resins can also be used in the hot melt compositions of the invention. The "average" number of epoxy groups per molecule is defined as the number of epoxy groups in the epoxy resin divided by the total number of epoxy molecules present.

Useful epoxy resins include those which contain cyclohexene oxide groups such as the epoxycyclohexane carboxylates, typified by 3,4-epoxycyclohexylmethyl-3,4-epoxycyclohexane carboxylate, 3,4-epoxy-2-methylcyclohexylmethyl-3,4-epoxy-2-methylcyclohexane carboxylate, and bis(3,4-epoxy-6-methylcyclohexylmethyl) adipate. For a more detailed list of useful epoxides of this nature, reference may be made to U.S. Pat. No. 3,117,099.

Further epoxy resins which are useful in the present invention include glycidyl ether monomers such as the glycidyl ethers of polyhydric phenols obtained by reacting a

polyhydric phenol with an excess of chlorohydrin such as epichlorohydrin, for example, the diglycidyl ether of 2,2-bis-(4-hydroxyphenyl)propane (Bisphenol A). Further examples of epoxides of this type are described in U.S. Pat. No. 3,018,262.

Many commercially available epoxy resins can be used in this invention. In particular, epoxides which are readily available include octadecylene oxide, epichlorohydrin, styrene oxide, vinylcyclohexene oxide, glycidyl methacrylate, diglycidyl ether of Bisphenol A, diglycidyl ether of Bisphenol F, vinylcyclohexene dioxide, 3,4-epoxycyclohexyl-methyl-3,4-epoxycyclohexene carboxylate (for example, having the trade designation "ERL-4221" from Union Carbide Corp.), 2-(3,4-epoxycyclohexyl-5,5-spiro-3,4-epoxy) cyclohexane-metadioxane, bis(3,4-epoxycyclohexyl) adipate, dipentene dioxide, epoxidized polybutadiene, epoxy silanes, for example, beta-3,4-epoxycyclohexylethyltrimethoxysilane and gamma-glycidoxypopyltrimethoxysilane, flame retardant epoxy resins, 1,4-butanediol diglycidyl ether, hydrogenated bisphenol A-epichlorohydrin based epoxy resins, and polyglycidyl ethers of phenol-formaldehyde novolaks.

The primer may additionally contain additives. Some primers include a multi-functional acrylate. For example, the primer may benefit from the inclusion of TMPTA (trimethylolpropane triacrylate) at moderate levels. The TMPTA may be added at about 1% to about 10% by weight based on the weight of the primer. TMPTA is especially beneficial for polyester tape backings.

The primer may also benefit from the addition of a fumed amorphous silica. This is especially beneficial when the primer is coated onto a backing and then rolled, so that the primer comes into contact with the unprimed surface of the backing. An example of a suitable fumed amorphous silica includes those sold under the tradename Aerosil OX50, commercially available from Degussa Corp., Ridgefield Park, NJ. Generally, the amount of silica added to the primer is dependent upon the coating conditions and the type of coater. In certain embodiments, the amount of silica is between 2 and 10% by weight, for example between about 3 and 8% based upon the solids of the primer coating solution. Too much silica yields a coating solution that is too viscous but a coating solution with too little silica yields a dry primer coating that may transfer to un-coated backing.

The primer of the present invention may also include fillers. Examples of fillers include, for example, nanoclay, flame retardants, plasticizers, tackifiers, fillers, colorants,

ultraviolet light stabilizers, antioxidants, processing aids (urethanes, silicones, fluoropolymers, etc.), electrically or thermally conductive fillers, magnetic fillers, pigments, antistatic agents, lubricants, wetting agents, surfactants, dyes, coupling agents, suspending agents, stabilizers and combinations thereof.

5

Making the Primer

The primer is made by mixing all components into a large volume holder and mechanically agitating the components until mixed. Generally, the maleated thermoplastic elastomer is added at about 1% to about 80 % by weight of the entire primer, for example between about 10% and about 50% by weight. The polyolefin is added at about 5% to about 80 % by weight, for example between about 30% and about 50% by weight. The tackifier is added at about 5% to about 60% by weight, for example between about 20% and about 40% by weight.

If a crosslinker is desired, the crosslinker is added to the other components in an amount between about 0.1% and about 3% by weight, for example between about 0.1% and about 1% by weight. If an epoxy is desired, the epoxy is added to the other components in an amount between about 0% and about 5% by weight, for example between about 1% and about 4% by weight.

The components are generally dissolved in a carrier solvent until the solution reaches about 10% solids. The mixture of components is then agitated by any mechanical means, for example a mechanical stirring device, at room temperature, or about 25°C. The temperature can be elevated to expedite blending, however, the temperature should not exceed the boiling point of the carrier solvent.

25 Tape

The primer can be applied to a substrate (e.g., tape backing) through any of a variety of processes including solution coating, solution spraying, emulsion coating, gravure coating, or a number of other such processes known to those skilled in the art. The primer can be used to promote the adhesion to metal, metal oxides, metal hydroxide, polyesters, polyamides, polyurethanes, polyvinyl alcohol, poly(ethylene-co-vinyl alcohol), other polar polymers and other polymer surfaces containing hydroxyl or amino groups. Backings of interest include, but are not limited to, polyesters, polyolefins, papers, foils,

poly(meth)acrylates, polyurethanes, perfluoropolymers, polycarbonates, ethylene vinyl acetates, and the like. Backings of vinyl films, woven and nonwoven sheets, woven and nonwoven fabrics, foam, papers, polymer coated papers and retroreflective sheeting may also be useful. In certain embodiments, the backings are films of polyolefins (e.g.,
5 polyethylene and propylene), especially corona-treated polyolefin films, polyethylene terephthalate and elastomer-saturated paper. In a specific embodiment, the backing is aluminum coated.

Useful coating weights of the primer range from about 0.1 to about 5 mg/cm². In some embodiments, the coating weight is from about 0.2 to about 1.0 mg/cm², and in
10 specific embodiments the coating weight is from about 0.3 to about 0.5 mg/cm². Once coated onto a backing, the primer layer may be dried. A drying step is generally performed at an elevated temperature, reduced pressure, or both. Generally, it is beneficial to crosslink the primer after the pressure sensitive adhesive has been coated.

In the tape construction of the present invention, a pressure sensitive adhesive,
15 generally a poly- α -olefin pressure sensitive adhesive is coated onto the primed surface of the tape backing (i.e., substrate) prior to being crosslinked. The pressure sensitive adhesive may be coated by any suitable process, such as knife coating out of solution or extrusion coating from the melt of the adhesive. The primer is used to increase the bond of the pressure sensitive adhesive to the tape backing so as to permit clean removal from
20 most surfaces. A primer's performance depends on various factors, for example the adherend, the temperature, the angle of peel and the rate of peel. Therefore, a careful balance must be found and even similar materials if compounded in different ratios may exhibit significantly different results depending on the exact conditions of the tests or applications.

25 The pressure sensitive adhesive and the primer are then crosslinked. Crosslinked means that a connection is formed within or between one or more polymer molecules wherein the connection is a covalent bond resulting from a chemical reaction. The tape article is exposed to source of activating radiation to crosslink the polymer components. In some embodiments, the tape article may be exposed to heat to crosslink the polymer
30 components. To achieve crosslinking, the crosslinking agent in the primer and the pressure sensitive adhesive composition may be exposed to actinic radiation thereby generating free radicals which promote the crosslinking process. Commercially available

actinic radiation sources include infrared, visible, and ultraviolet radiation. The use of UV radiation is explained in Hoyle and Kinstle, Radiation Curing of Polymeric Materials, (American Chemical Society, 1990).

Generally, the wavelength range to activate the radiation activatable crosslinking agents is about 250 nm to about 400 nm. The dose of radiant energy applied in this wavelength range is generally less than about 1500 millijoules/cm², for example less than about 800 millijoules/cm². In specific embodiments, the dose of radiant energy required to crosslink the primer and the pressure sensitive adhesive is less than about 500 millijoules/cm², for example about 300 millijoules/cm². Details of a radiation crosslinking process are disclosed in U.S. Pat. Nos. 4,181,752 and 4,329,384.

Objects and advantages of this invention are further illustrated by the following examples. The particular materials and amounts thereof, as well as other conditions and details, recited in these examples should not be used to unduly limit this invention.

Examples

Table of Materials

Name	Material	Supplier
Acrylate	Terpolymer consisting of iso-octyl acrylate/ N-vinylcaprolactam/ acrylic acid	Prepared as described in Example 1 of U.S. Patent No. 5,677,376 to Groves.
Elastomer	Maleated S-EB-S copolymer	Commercially available as KRATON FG 1901X, from Kraton Polymers, Houston, TX.
Resin-1	A hydrogenated hydrocarbon resin.	Commercially available as REGALREZ 1139, from Hercules, Inc., Wilmington, DE.
Resin-2	A hydrogenated hydrocarbon resin.	Commercially available as REGALREZ 1126, from Hercules, Inc., Wilmington, DE.
Resin-3	Saturated hydrocarbon Resin	Commercially available as ARKON P-115 from Arakawa Chemical Industries, Osaka, Japan.

Name	Material	Supplier
Clay	An organophilic surface treated clay	Commercially available as CLOISITE 20A, from Southern Clay Products, Gonzalez, TX.
CP	Chlorinated Polyolefin	Commercially available as Chlorinated Polyolefin 343-3 (25% solids in Xylene), from Eastman Chemical Co., Kingsport, TN.
CH	Cyclohexane	Commercially available from Aldrich Chemical Co. (Milwaukee, WI).
P-6	Polyhexene	Made as described in Example 4 of U.S. Patent No. 5,644,007 to Davidson, et al. having an inherent viscosity of 2.2dL/g.
P-8	Poly-1-octene	Made as described Example 3 of U.S. Patent No. 5,644,007 to Davidson, et al. having an inherent viscosity of 2.5dL/g.
XL-1	2,4-bis(trichloromethyl)-6-4'-methoxyphenyl-sym-triazine	Made by the co-trimerization of an aryl nitrile with trichloroacetonitrile in the presence of HCl gas and a Lewis acid such as AlCl ₃ , AlBr ₃ , etc. [Bull. Chem. Soc. Japan. Vol. 42, page 2924 (1969)].
XL-2	t-butylanthraquinone	Commercially available from Aldrich Chemical Co., Milwaukee, WI.
XL-3	Neutralized salt of 1,8-diazabicyclo[5.4.0]undec-7-ane	Commercially available as U-Cat CA-102 from Sun Apollo, Japan.
Eth	Anhydrous Ethanol	Commercially available from Aldrich Chemical Co. (Milwaukee, WI).
Epoxy-1	Epoxy Resin	Obtained as ERL-4221 from Union Carbide Corp., Danbury, CT.

Name	Material	Supplier
Epoxy-2	Bisphenol A type epoxy resin	Commercially available from Yuka Shell, K.K.
Epoxy-3	Bisphenol A /Epichlorohydrin based Epoxy Resin	Commercially available under the tradename EPON Resin 828, from Shell Oil Co., Houston, TX.
TMPTA	Trimethylolpropane triacrylate	Commercially available from Sartomer Co., Exton, PA
Irganox 1076	A hindered phenol type antioxidant. Octadecyl 3,5-bis(1,1-dimethylethyl)-4-hydroxybenzene-propanoate.	Commercially available under the tradename IRGANOX 1076 from CIBA-GEIGY Corp., Tarrytown, NY
Irganox B561	A 4:1 blend of 4 parts tris(2,4-di-tert-butylphenyl)phosphite CAS No. [31570-04-4] to 1 part 3,5-bis(1,1-Dimethylethyl)-4-hydroxybenzenepropanoic acid, 2,2-bis[[3-[3,5-bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-oxopropoxy]methyl]1,3-propanediyl ester CAS No. [6683-19-8]	Commercially available under the tradename IRGANOX B561 from CIBA-GEIGY Corp., Tarrytown, NY
X	Xylene	Commercially available from Aldrich Chemical Co. (Milwaukee, WI).
Silane	beta-(3,4-epoxycyclohexyl)ethyltrimethoxy silane	Commercially available under the tradename SILQUEST A-186 from Crompton Corp., South Charleston, WV.
TTE	Triethanolamine titanate	Commercially available under the tradename TYZOR TE from E.I. du Pont and de Nemours and Co., Wilmington, DE.
PSA-1	poly- α -olefin pressure sensitive adhesive	Made as described in Synthesizing PSA-1.
PSA-2	poly- α -olefin pressure sensitive adhesive	Made as described in Synthesizing PSA-2.
PSA-3	poly- α -olefin pressure sensitive adhesive	Made as described in Synthesizing PSA-3.

Name	Material	Supplier
PSA-4	poly- α -olefin pressure sensitive adhesive	Made as described in Synthesizing PSA-4.
PSA-5	poly- α -olefin pressure sensitive adhesive	Made as described in Synthesizing PSA-5.

Synthesizing PSA-1

The PSA-1 formulation included 81.875% (by weight) P-8, 18% (by weight) Resin-3, 0.125% (by weight) XL-1. The materials were mixed with a Brabender Plasti-corder (commercially available from C.W. Brabender Instruments, Inc., Hackensack, NJ) in an attached 400ml mixing bowl at 175°C for 10 minutes.

Synthesizing PSA-2

PSA-2 was made as PSA-1 except using the following formulation: 84.8% P-6, 15% Resin-1, 0.2% XL-2.

Synthesizing PSA-3

PSA-3 was made as PSA-1 except using the following formulation: 100 parts by weight P-8, 21.5 parts by weight Resin-2, 1.5 parts by weight Irganox 1076, 3.0 parts by weight Irganox B561, 0.3 parts by weight XL-1 and 7.2 parts by weight TMPTA.

Synthesizing PSA-4

PSA-4 was made as PSA-1 except with the following formulation: 100 parts by weight P-8, 42.9 parts by weight Resin-2, 1.5 parts by weight Irganox 1076, 3.0 parts by weight Irganox B561, 0.3 parts by weight XL-1, and 7.2 parts by weight TMPTA.

Synthesizing PSA-5

PSA-5 was made as PSA-1 except using the following formulation: 84.8% P-8, 15% Resin-2, 0.2% XL-1.

Testing Methods

Shear is measured from stainless steel in minutes by ASTM D6463-99, with the exception that the tape sample measures 1 inch by 1 inch and weighs 1000 grams.

5 Adhesive Transfer was estimated by visual inspection of the shear panel and the tape and represents the percentage by area of adhesive that transferred from the backing to the shear panel.

Removal Force is measured from anodized aluminum. The test procedure is to apply a 0.5 inch wide sample of tape to an aluminum panel that has been anodized in chromic acid and left unsealed. The tape sample is removed by peeling from the
10 aluminum panel at a peel angle of 90 degrees and at a peel rate of 12 inches per minute using any of the many common peeling devices such as an Instron. Two pieces of information are reported from this test the removal force to remove the 0.5 inch sample and the failure mode. There are three modes of failure: "adhesive", "cohesive" and "interfacial". "Adhesive" is the separation of the adhesive and the adherend at the
15 adhesive adherend interface. "Cohesive" is failure in the bulk of the adhesive. "Interfacial" refers to failure at the interface between the adhesive and the backing. The failure mode is determined by visual and tactile inspection. If the tape is removed and there is a visible deposit of adhesive on the panel and it feels tacky and the tape backing looks and feels adhesive free then that is reported as interfacial failure. If the tape is
20 removed and there is a visible deposit of adhesive on the panel and it feels tacky and the tape backing looks and feels tacky then the failure mode is cohesive. If the tape is removed and there is no visible deposit of adhesive on the panel and the tape backing looks and feels adhesive coated then the failure mode is adhesive.

25 Primers 1-6

A series of primers were created using the formulations shown in Table 1. The components are listed in their weight percentage. The components were blended in the weight percentages shown in toluene to achieve a 10% solids solution until they were dissolved.

30

Table 1

Primer	Elastomer	P-6	P-8	Resin-1	Resin-3	Epoxy	XL-1	XL-2
Primer 1	32.7	0	21.8	43.6	0	1.6	0	0.3
Primer 2	32.7	0	21.8	0	43.6	1.6	0	0.3
Primer 3	32.7	21.8	0	43.6	0	0	0	0.3
Primer 4	32.7	21.8	0	43.6	0	1.6	0	0.3
Primer 5	32.7	21.8	0	0	43.6	1.6	0	0.3
Primer 6	20	44	0	34	0	1.6	0.3	0

Examples 1-6 and Comparative Examples A and B

A series of tapes were formed by coating a primer solution as detailed in Table 2 on to C83490 SBR-saturated paper tape backings (Kimberly-Clark Co.; Roswell, Ga.).

The primer coated papers were then dried at 160°F (71°C) for 10 minutes and the primer was then coated with PSA-1. PSA-1 was coated using a Haake Rheocord (commercially available from Haake, Inc., Saddlebrook, NJ) with an attached 0.75 inch (1.9 cm) single screw extruder and a 5.25 inch (13.3 cm) extrusion die at a coating weight of 55 grams/m².

The PSA-1 coated papers were rolled up with a silicone liner. After 2 hours, the PSA-1 coated papers were cured under a nitrogen atmosphere with 300 mJ/cm² of energy as measured by a sensing device (commercially available under the tradename UVIMAP 365 from Electronic Instrumentation and Technology, Inc., Sterling, Va.) from medium pressure Hg lamps. (Calibration standard for UV energy was MIL-STD-45662A.)

Table 2

Example	Primer
1	Primer 1
2	Primer 2
3	Primer 3
4	Primer 4
5	Primer 5
6	Primer 6
Comp. Ex. A	None
Comp. Ex. B	Primer as described in Example 2 of U.S. Patent No. 5,846,653, to Hawkins.

Examples 1-6 and Comparative Examples A and B were tested according to the Failure Test Method as references above. The results are shown in Table 3.

Table 3

Example	Shear (min)	Adhesive Transfer (%)	Removal Force oz/0.5inch	Failure Mode
1	298.6	5	52	Cohesive
2	286	5	53	Cohesive
3	332.4	0	54	Cohesive
4	218.5	0	52	Cohesive
5	249.1	5	48	Cohesive
6	492.3	0	45	Cohesive
Comp. Ex. A	271.7	10	38	Interfacial
Comp. Ex. B	304.4	30	38	Interfacial

These results indicate that the primer of the invention maintains shear strength while increasing removal force and avoiding interfacial failure.

5

Example 7 and Comparative Examples C and D

A series of primers were formulated as shown in Table 4, and made as described for Primers 1-6. The components are listed in their weight percentage.

10

Table 4

Primer	Elastomer	P-6	P-8	Resin-1	Epoxy-1	Epoxy-2	XL-1	XL-3
Primer 7	20	44	0	34	1.6		0.3	0
Primer C	94.4	0	0	0	0	4.7	0	0.9
Primer D	94.4	0	0	0	0	4.7	0.9	0

Example 7 and Comparative Examples C-E were made as in Examples 1-6 with the Primer as detailed in Table 5. Comparative Example F was made as Example 7 with the exception that the primer was cured prior to the application of the adhesive.

15

Table 5

Example	Primer
7	Primer 7
Comp Ex. C	Primer C
Comp. Ex. D	Primer D
Comp. Ex. E	None
Comp Ex. F	Primer 7

Example 7 and Comparative Examples C -F were tested according to the Failure Test Method as references above. The results are shown in Table 6.

Table 6

Example	Shear (min)	Adhesive Transfer (%)	Removal Force oz/0.5inch	Failure Mode
7	492.3	0	45	Cohesive
Comp. Ex. C	513.2	60	33	Interfacial
Comp. Ex. D	402.2	60	27	Interfacial
Comp. Ex. E	501.9	50	30	Interfacial
Comp. Ex. F	442.8	50	27	Interfacial

5

These results indicate that the primer of the invention maintains shear strength while increasing removal force and avoiding interfacial failure. These results also indicate that the tapes of the invention benefit from curing the primer and the adhesive together.

10 Examples 8 – 10 and Comparative Examples G - I

A series of primers were formulated as shown in Table 7, and made as described for Primers 1-6. The components are listed in their weight percentage.

Table 7

Primer	Elastomer	P-6	Resin-1	Epoxy-1	XL-1
Primer 8	16.4	38.1	43.6	1.6	0.3
Primer 9	27.2	27.2	43.6	1.6	0.3
Primer 10	38.1	16.4	43.6	1.6	0.3
Primer G	0	54.5	43.6	1.6	0.3
Primer H	54.5	0	43.6	1.6	0.3

15

Examples 8-10 and Comparative Examples G and H were prepared by dissolving the primer as detailed in Table 8 in toluene at room temperature to prepare primer solution with 15% solids. The primer solution was coated with a knife coater on aluminum vapor coated polyethyleneterephthalate (commercially available from Courtaulds Performance Films, Martinsville, Virginia) backing and the dry thickness was controlled at 0.1 to 0.3 mil (2.5 – 7.6 micrometer). The primer coated polyethyleneterephthalate were then dried at 160°F (71°C) for 10 minutes.

20

The tape was made by coating hot-melt PSA-2 to the primed backing followed by UV crosslinking with a medium pressure mercury vapor lamp at about 500 milli joules dosage (commercially available from UVEXS Incorp., Sunnyvale, CA). The thickness of PSA is typically controlled at 2 mil (50.4 micrometer).

5

Table 8

Example	Primer
8	Primer 8
9	Primer 9
10	Primer 10
Comp. Ex. G	Primer G
Comp. Ex. H	Primer H
Comp. Ex. I	None

The resulting tapes were tested according to the Failure Test Method as references above. The results are shown in Table 9.

10

Table 9

Example	Removal Force oz/0.5inch	Failure	Removal Force oz/0.5inch (after humidity aging and 70% Relative Humidity and at 80 °C)	Failure (after humidity aging)
8	50.2	Cohesive	51.0	Adhesive
9	54.8	Cohesive	50.8	Adhesive
10	56.8	Cohesive	52.0	Adhesive
Comp. Ex. G	30.6	Interfacial	31.3	Interfacial
Comp. Ex. H	48.0	Interfacial	46.8	Interfacial
Comp. Ex. I	18.6	Interfacial	17.7	Interfacial

These results indicate that the addition of the Elastomer to the primer composition improves the adhesion to the film backing. Additionally, the addition of a polyhexene component enhances the bonding of the primer to PSA-2.

15

Examples 11 – 15 and Comparative Examples J and K

A series of primers were formulated as shown in Table 10, and made as described for Primers 1-6. The components are listed in their weight percentage.

20

Table 10

Primer	Elastomer	P-6	Resin-1	Epoxy-1	XL-1
Primer J	57.9	38.6	0	2.9	0.6
Primer 11	48.5	32.4	16.2	2.4	0.5
Primer 12	41.8	27.9	27.9	2.1	0.4
Primer 13	36.7	24.4	36.7	1.8	0.4
Primer 14	32.7	21.8	43.6	1.6	0.3
Primer 15	29.5	19.6	49.1	1.5	0.3

Examples 11-13 and Comparative Examples J-K were prepared as examples 8-10 and Comparative Examples G and H using the primer as detailed in Table 11.

5

Table 11

Example	Primer
11	Primer 11
12	Primer 12
13	Primer 13
14	Primer 14
15	Primer 15
Comp. Ex. J	Primer J
Comp. Ex. K	None

The resulting tapes were tested according to the Failure Test Method as references above. The results are shown in Table 12.

10

Table 12

Example	Removal Force oz/0.5inch	Failure
11	49.6	Mixed Cohesive and Interfacial
12	61.3	Cohesive
13	60.1	Cohesive
14	67.87	Cohesive
15	51.8	Mixed Cohesive and Interfacial
Comp. Ex. J	42.0	Interfacial
Comp. Ex. K	18.6	Interfacial

These results indicate that the loading of the resin has an effect on the performance of primer.

Examples 16-19

- 5 Primer 16 was formulated as shown in Table 13 with all amounts shown in percent by weight. The formulation was dissolved in toluene to achieve a primer solution with 15% solids.

Table 13

Primer	Elastomer	P-6	Resin-1	Epoxy-1	XL-1
Primer 16	32.7	21.8	43.6	1.6	0.3

10

Primer 16 was blended with a fumed amorphous silica (commercially available as Aerosil OX50 from Degussa Corp., Ridgefield Park, NJ), dispersed in toluene to 10% solids, in the amounts shown in Table 14.

15

Table 14

Primer	Primer 16 in toluene	Silica in toluene
Primer 17	1000g	1.5g
Primer 18	1000g	3.0g
Primer 19	1000g	4.5g

20

Examples 16-19 were prepared by coating the primer with a knife coater as detailed in Table 15 on aluminum vapor coated polyethyleneterephthalate (commercially available from Courtaulds Performance Films, Martinsville, Virginia) backing and the dry thickness was controlled at 0.1 to 0.3 mil (2.5 – 7.6 micrometer). The primer coated polyethyleneterephthalate films were then dried at 160°F (71°C) for 10 minutes.

Table 15

Example	Primer
Example 16	Primer 16
Example 17	Primer 17
Example 18	Primer 18
Example 19	Primer 19

The tapes of Example 16 – 19 were tested by rolling the primed backing so that the primed surface was in contact with an un-primed surface. The wound roll of primed backing was then unwound. Blocking occurs when the primer transfers to the un-primed surface of the backing upon unwind. The amount of blocking was observed visually and is detailed in Table 16. “Severe” blocking was observed when more than 30% of the primer appeared to have transferred to the unprimed surface.

Table 16

Example	Blocking
Example 16	Severe
Example 17	Less than 5%
Example 18	No Blocking
Example 19	No Blocking

Examples 20-23 and Comparative Examples L and M

A series of primers were formulated as shown in Table 17, and made as described for Primers 1-6. The components are listed in their weight percentage.

Table 17

Primer	Elastomer	XL-2	P-6	Resin-1	Epoxy-1	Modifier
20	32.7	0.3	21.8	43.6	1.6	None
21	32.5	0.3	21.7	43.3	1.6	TMPTA 0.5
22	31.6	0.3	21.1	42.2	1.6	Clay 3.2

The primers evaluated were coated out of solvent using a ground glass rod with a 223g weight on either end to spread the solution evenly on a 0.002 inch thick polyester film (commercially available under the tradename SCOTCH PAR P-0862197 Polyester Film from 3M Company, Saint Paul, MN) using the primer as detailed in Table 18. These coated films were then dried in a 250°F oven for 5 minutes. The primed films were then adhesive coated with the pressure sensitive adhesive formulation as shown in Table 18. The pressure sensitive adhesive was extruded onto the primed backing to form a tape. The tapes were rolled up with a silicone liner (commercially available under the tradename

SCW106 NP from 3M Company) before exposure to UV radiation. The silicone release liner was removed and the tape was cured under a nitrogen atmosphere with 300 mJ/cm² of energy as measured by a UVIMAP™ 365 sensing device (Electronic Instrumentation and Technology, Inc., Sterling, VA) from medium pressure mercury lamps.

5

Table 18

Example	Primer	Pressure Sensitive Adhesive
Example 20	Primer 20	PSA-3
Example 21	Primer 21	PSA-3
Example 22	Primer 22	PSA-3
Example 23	Primer 20	PSA-4
Example 24	Primer 21	PSA-4
Example 25	Primer 22	PSA-4
Comp. Ex. L	None	PSA-3
Comp. Ex. M	None	PSA-4

The tapes were tested by adhering these onto anodized aluminum panels. The tapes were then peeled by hand at 180° and 90° angles and the failure mode noted as adhesive, cohesive, or interfacial as defined above. In the event that there was interfacial failure on as little as 5% of the tape the failure was still designated as interfacial. The results are shown in Table 19.

10

Table 19

Example	180 degree	90 degree
Example 20	Cohesive	Cohesive
Example 21	Cohesive	Cohesive
Example 22	Cohesive	Cohesive
Example 23	Cohesive	Interfacial
Example 24	Cohesive	Cohesive
Example 25	Cohesive	Cohesive
Comp. Ex. L	Cohesive	Interfacial
Comp. Ex. M	Interfacial	Cohesive

15

Comparative Example N

Primer N was formulated as shown in Table 20 with all amounts shown in percent by weight. The formulation was dissolved in toluene to achieve a primer solution with 15% solids.

Table 20

Primer	Elastomer	CH	Eth	CP	E3	X	AI	Silane	TTE
Primer N	0.66	47.97	8.74	5.3	0.44	30.6	5.55	0.44	0.3

Comparative Example N was prepared by dissolving the primer as detailed in Table 20 in toluene at room temperature to prepare primer solution with 15% of solid.

The primer solution was coated with a knife coater on aluminum vapor coated polyethyleneterephthalate (commercially available from Courtaulds Performance Films, Martinsville, Virginia) backing and the dry thickness was controlled at 0.1 to 0.3 mil (2.5 – 7.6 micrometer). The primer coated papers were then dried at 160°F (71°C) for 10 minutes.

The tape was made by coating hot-melt PSA-5 to the primed backing followed by UV crosslinking with a medium pressure mercury vapor lamp at about 500 milli joules dosage (commercially available from UVEXS Incorp., Sunnyvale, CA). The thickness of PSA is typically controlled at 2 mil (50.4 micrometer).

The resulting tape was tested for 90 degree peel at 12 inches/minute from stainless steel. The tape was tested both at 1 minute dwell and after 3 days at 70 °C. For both time intervals, the tape failed with 100% interfacial failure.

Various modifications and alterations that do not depart from the scope and spirit of this invention will become apparent to those skilled in the art. This invention is not to be unduly limited to the illustrative embodiments set forth herein.

What is Claimed is:

1. A primer comprising:
 - (a) a maleated thermoplastic elastomer;
 - (b) a non-halogenated polyolefin; and
 - (c) a resin.
2. The primer of claim 1 comprising a crosslinking agent.
3. The primer of claim 2 wherein the crosslinking agent is 2,4-bis(trichloromethyl)-6-4'-methoxyphenyl-sym-triazine.
4. The primer of claim 1 comprising an epoxy.
5. The primer of claim 1 wherein the maleated thermoplastic elastomer is a maleated rubber.
6. The primer of claim 5 wherein the rubber is a block copolymer.
7. The primer of claim 6 wherein the block copolymer is a styrene-block-ethylene-co-butene-block-styrene block copolymer.
8. The primer of claim 1 wherein the non-halogenated polyolefin comprises a material selected from polyhexene, polyoctene, or combinations thereof.
9. The primer of claim 1 wherein the resin is a hydrogenated hydrocarbon resin.
10. The primer of claim 1 comprising fumed amorphous silica.
11. The primer of claim 1 comprising trimethylolpropane triacrylate.

12. A tape comprising:
- (a) a substrate;
 - (b) a primer coated on at least one surface of the substrate, the primer comprising a maleated rubber, a non-halogenated polyolefin and a resin; and
 - (c) a pressure sensitive adhesive coated on the primer.
13. The tape of claim 12 wherein the primer comprises a crosslinking agent.
14. The tape of claim 12 wherein the primer comprises an epoxy.
15. The tape of claim 12 wherein the polyolefin is selected from polyhexene, polyoctene, or combinations thereof.
16. The tape of claim 13 wherein the crosslinking agent is 2,4-bis(trichloromethyl)-6-4'-methoxyphenyl-sym-triazine.
17. The tape of claim 12 wherein the pressure sensitive adhesive is a poly- α -olefin pressure sensitive adhesive.
18. The tape of claim 17 wherein the poly- α -olefin pressure sensitive adhesive is crosslinked.
19. The tape of claim 12 wherein the substrate is a saturated paper.
20. The tape of claim 12 wherein the substrate is a polymer film.
21. The tape of claim 12 wherein the substrate comprises a material selected from polyesters, polyolefins, papers, foils, polyacrylates, polyurethanes, perfluoropolymers, polycarbonates, ethylene vinyl acetates, vinyl, fabrics, foam, polymer coated papers, retroreflective sheeting and combinations thereof.
22. The tape of claim 20 wherein the polymer film comprises polyethylene terephthalate.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 02/28101

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C09D153/02 C08K5/3492 C09J7/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C09D C08K C09J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 6 048 610 A (TYNAN JOHN ET AL) 11 April 2000 (2000-04-11) column 1, paragraph 1 column 2, paragraph 4 claims 1,3,8-10,12; examples ---	1,2,5-7, 12,13, 20,21
X	US 3 970 771 A (DAVISON SOL) 20 July 1976 (1976-07-20) column 2, line 46 - line 57; examples ---	1
A	US 5 846 653 A (HAWKINS STEPHEN J) 8 December 1998 (1998-12-08) cited in the application claims --- -/-	1,12,17, 19

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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- *Z* document member of the same patent family

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INTERNATIONAL SEARCH REPORT

In International Application No.

PCT/US 02/28101

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Information on patent family members

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PCT/US 02/28101

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